

## CLAIMS

1. A method for thermogravimetrically testing the behaviour of a solid material in the presence of a controlled gaseous atmosphere, wherein:
  - 5 - a plurality of samples (10) are placed in the presence of said gaseous atmosphere inside the same controlled-atmosphere furnace (4),
  - each sample has its own associated balance (38) having an error of less than 100  $\mu\text{g}$ ,
  - 10 - the samples (10) are subjected to successive predetermined thermal cycles each comprising a heating step, during which the samples are heated directly, and a cooling step, during which the samples are not heated,
  - the weight of each sample is measured and recorded independently, in a continuous manner, at least during a predetermined period during the heating step of each thermal cycle.
- 15 2. The method as claimed in claim 1, wherein the weight of each sample (10) is measured and recorded continuously at least during a high-temperature stage of the heating step of each thermal cycle.
- 20 3. The method as claimed in either claim 1 or claim 2, wherein, in each thermal cycle, the samples (10) are heated so that their temperature is from 400°C to 1800°C at least during a high-temperature stage of the heating step.
- 25 4. The method as claimed in any one of claims 1 to 3, wherein, in each thermal cycle, the samples (10) are heated so that their temperature is greater than 1100°C at least during a high-temperature stage of the heating step.

5. The method as claimed in any one of claims 1 to 4, wherein, in each thermal cycle, the samples (10) are heated at a rate of heating greater than 300°C/minute.
- 5 6. The method as claimed in any one of claims 1 to 5, wherein, in each thermal cycle, the samples (10) are cooled at a rate of cooling greater than 100°C/minute.
- 10 7. The method as claimed in any one of claims 1 to 6, wherein the samples (10) are subjected to thermal cycles each comprising a heating step, which consists of a phase of rise in temperature having a duration of less than 5 minutes and a high-temperature stage having a duration of the order of 60 minutes, and a cooling step, which consists of a phase of fall in temperature having a duration of less than 10 minutes and a low-temperature stage having a duration of from 0 to 15 minutes.
- 15 8. The method as claimed in any one of claims 1 to 7, wherein the samples (10) are subjected to a number of successive thermal cycles of from 10 to 3000.
- 20 9. A device for thermogravimetrically testing the behaviour of a solid material in the presence of a controlled gaseous atmosphere, comprising:
- a furnace (4) having a controlled gaseous atmosphere,
  - means (6) for weighing the material placed in the furnace, having an error of less than 100 µg,
  - 25 - confining means (7, 8, 34) suitable for limiting any disturbance to the weighing means owing to the external environment of the device and/or the controlled gaseous atmosphere,
- wherein
- the furnace (4) is suitable for receiving a number N, which is strictly greater than 1, of samples (10) of the material,
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- the furnace comprises means (11) for heating the samples directly, which means are capable of subjecting the samples to successive predetermined thermal cycles each comprising a heating step, during which the samples are heated, and a cooling step, during which the samples are not heated,
  - 5 - the weighing means comprise N independent balances (38) having an error of less than 100  $\mu$ g, each balance being capable of measuring and recording the weight of a sample continuously at least during a predetermined period during the heating step of each thermal cycle,
  - the device has a star-shaped structure overall.
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10. The device as claimed in claim 9, wherein each balance (38) is capable of measuring and recording the weight of a sample (10) continuously at least during a high-temperature stage of the heating step of each thermal cycle.
- 15 11. The device as claimed in either claim 9 or claim 10, having a star-shaped structure overall, in which at least the balances are arranged in the shape of a star.
12. The device as claimed in claim 11, wherein its star-shaped structure is suitable  
20 for receiving the samples close to one another in a central portion of the furnace.
13. The device as claimed in any one of claims 9 to 12, wherein the direct heating means (11) are capable of bringing the samples to a temperature greater than  
25 1100°C.
14. The device as claimed in any one of claims 9 to 13, wherein the direct heating means (11) are capable of heating the samples at a rate of heating greater than  
30 300°C/minute.

15. The device as claimed in any one of claims 9 to 14, wherein the direct heating means (11) are capable of cooling the samples at a rate of cooling greater than 100°C/minute.
- 5 16. The device as claimed in any one of claims 9 to 15, wherein the direct heating means (11) are capable of carrying out thermal cycles each comprising a heating step, which consists of a phase of rise in temperature having a duration of less than 5 minutes and a high-temperature stage having a duration of the order of 60 minutes, and a cooling step, which consists of a phase of fall in temperature  
10 having a duration of less than 10 minutes and a low-temperature stage having a duration of from 0 to 15 minutes.
17. The device as claimed in any one of claims 9 to 16, wherein the direct heating means (11) are capable of carrying out more than 3000 successive thermal  
15 cycles.
18. The device as claimed in any one of claims 9 to 17, wherein the furnace comprises at least N high-radiation lamps (11), a chamber (9) for receiving the samples, made of a thermal resistant material that is transparent to the radiation  
20 of the lamps, and a reflective peripheral inner face (12) having a shape that is suitable for defining at least N separate zones of maximum illumination inside the chamber, at the site of which the samples may be placed.
19. The device as claimed in claim 18, wherein the peripheral inner face (12) of the  
25 furnace forms at least N ellipse portions arranged in a star, each ellipse having a first focus (13) outside the chamber (9), called the emitting focus, at the site of which there is arranged a lamp, and a second focus (14) inside the chamber, called the receiving focus, at the site of which a sample may be placed, at least N of said ellipses having separate receiving focuses.

20. The device as claimed in claims 12 and 19, wherein the chamber (9) and the receiving focuses (14) are situated in the central portion of the furnace and the emitting focuses (13) are situated in the peripheral portion of the furnace, and wherein the chamber (9) has reduced radial dimensions.
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21. The device as claimed in claims 9 to 20, wherein each balance (38) has an error of less than 10  $\mu\text{g}$ .
22. The device as claimed in any one of claims 9 to 21, wherein each balance (38) has a drift of less than 1  $\mu\text{g/h}$ .
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23. The device as claimed in any one of claims 9 to 22, wherein the balances (38) are mounted on the same support plate (3).
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24. The device as claimed in any one of claims 9 to 23, wherein the balances (38) are arranged above the furnace and each comprise a balance arm (39), means (40) for measuring a displacement or a force to which the balance arm is subjected, and a suspension rod (41) of aluminium which extends substantially vertically and has a lower end provided with a hook (49) for the attachment of a sample (10) and an upper end articulated with or fixed to a longitudinal end (45) of the balance arm, called the measuring end.
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25. The device as claimed in claims 12 and 24, wherein the balance arms (39) of the N balances are arranged in the shape of a star, each balance arm extending substantially according to a radial direction so that its measuring end (45) hangs over the central portion of the furnace.
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26. The device as claimed in either claim 24 or claim 25, wherein the suspension rods (41) are of the capillary type having two channels in order to permit the passage of thermocouple wires (48).
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27. The device as claimed in any one of claims 9 to 25, which comprises means (60) for supporting at least one piece of material (57), called a control, which means are suitable for holding the control in the immediate proximity of a sample (10) and are equipped with means (58) for measuring the temperature inside the control.
28. The device as claimed in claim 27, which comprises means (60) for supporting N controls, which means are suitable for holding a control beneath each sample, on its receiving focus, and are equipped with means (58) of the thermocouple wire type which end inside the control, for measuring independently the temperature of each of the controls.
29. The device as claimed in any one of claims 24 to 28, wherein the measuring means of at least one balance comprise an electronic weighing cell (40) to which the balance arm (39) is fixed.
30. The device as claimed in any one of claims 9 to 29, wherein the furnace (4) is mounted to slide according to a substantially vertical direction between a bottom preparation position, in which it is located beneath the lower end of the suspension rods (41) in order to allow samples to be attached and/or removed, and a top test position, in which the lower end of the suspension rods (41) extends inside the chamber (9) of the furnace.
31. The device as claimed in any one of claims 9 to 30, wherein the confining means comprise an upper protective bell (8) which is suitable for covering all of the balances (38) and for being fixed in a removable and air-tight manner to the support plate (3).

32. The device as claimed in any one of claims 9 to 31, wherein the confining means (7) comprise a confinement column between the support plate (3) and the furnace (4), which column is suitable for producing, on the one hand, an air-tight and removable connection, allowing the suspension rods to pass and be confined, between the support plate and the chamber of the furnace, and, on the other hand, an air-tight connection, by means of branches (29, 31), to means for generating the controlled gaseous atmosphere.
33. The device as claimed in any one of claims 9 to 32, wherein the means for generating the controlled gaseous atmosphere comprise, on the one hand, a vacuum pump and a gas inlet pipe (28) which are each connected to a branch (29) of the confinement column, and, on the other hand, a gas outlet pipe (27) which opens at a lower face of the chamber (9) of the furnace.
34. The device as claimed in any one of claims 9 to 33, wherein the confining means comprise means (34, 36, 37) for limiting gaseous and thermal exchanges between the furnace and the weighing means, said limiting means comprising a plurality of superposed and distant plates (34) which are integrated into the confinement column (7) above the branches thereof and which delimit a plurality of successive cooling chambers (55), each plate being pierced with N holes (35) for the passage of the suspension rods.
35. The device as claimed in claim 34, wherein each plate (34) has faces of low emissivity.
36. The device as claimed in claims 24 and 31, wherein each balance comprises a permanent counterweight (56) fixed to one longitudinal end (44) of the balance arm, called the calibrating end, so as to be suspended inside the protective bell (8).

37. The device as claimed in any one of claims 9 to 36, wherein the furnace (4) comprises temperature-regulating means of the PID type.
38. The device as claimed in any one of claims 9 to 37, wherein the furnace comprises temperature-regulating means suitable for controlling each lamp (11) independently.